SYSTEM AND METHOD FOR DETERMINING
MATERIAL PROPERTIES OF SAMPLES

CROSS-REFERENCE TO RELATED APPLICATIONS

This application claims priority to U.S. Provisional Patent Application Serial No. 60/401,186, filed August 5, 2002, which is hereby incorporated herein in its entirety by reference.

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates to a system and method for determining material properties and, more particularly, to a system and method for the determining the specific gravity, the density, and the absorption of uncompacted bituminous paving mixtures, as well as the bulk and absolute specific gravities and absorption properties of soils and aggregates, and the bulk specific gravity, permeability, and porosity of field cores and laboratory prepared specimens of compacted bituminous material.

Description of Related Art

In the construction industry, knowledge of the physical characteristics of the materials to be used during various phases of the construction process is often required. Among these materials, for example, are soils, aggregates and bituminous paving mixtures. The absolute (apparent), maximum, and bulk densities (or specific gravities) of these materials, as well as the absorption thereof, generally comprise material characteristics of common interest. The determination of these values for both coarse and fine aggregates can be time consuming, since the measurement procedure often requires the test sample to be soaked in a water bath for a period of 24 hours. Such measurement

methods and procedures can be found in, for example, ASTM Standard Nos. C128 – 97, *Standard Test Method for Specific Gravity and Absorption of Fine Aggregate* and C127 – 88, *Standard Test Method for Specific Gravity and Absorption of Coarse Aggregate*, which are incorporated herein by reference.

5   The standard method for determining the theoretical maximum specific gravity and density of bituminous paving mixtures, also referred to herein as the "Rice method" and the result of which is referred to herein as the "Rice value", involves the use of a system comprising a water-filled pycnometer operating in conjunction with a vacuum pump and associated lines, and a mass balance. The details of this method can be found

10   in ASTM Standard No. D2041-00, *Standard Test Method for Theoretical Maximum Specific Gravity and Density of Bituminous Paving Mixtures*, which is also incorporated herein by reference. In asphalt paving, the Rice value for a bituminous paving mixture is commonly used as the benchmark against which the density of field compacted material is compared. However, the Rice method may be cumbersome, time-consuming, subject

15   to inaccuracies, and destructive with respect to the sample since the necessary measurements are conducted with the sample immersed in water.

      Thus, there exists a need for a system and method capable of nondestructively determining the specific gravity, absorption, and other properties of soils, aggregates, and bituminous paving mixtures. Such a system and method should desirably be capable of

20   expeditiously producing the analysis of the sample, with minimal handling thereof, to obtain the necessary results with improved precision over commonly used methods.

BRIEF SUMMARY OF THE INVENTION

      The above and other needs are met by the present invention which, in one

25   embodiment, provides a method for nondestructively determining a material property of a porous sample. A first vessel is evacuated to a sub-atmospheric pressure, while a test pressure is established in a second vessel having the sample disposed therein, and wherein the test pressure is greater than the sub-atmospheric pressure. The pressures of the first and second vessels are then equalized by opening a valve mechanism operably

30   engaged between the first and second vessels. Each of the first and second vessels thereby experience a pressure change, wherein the pressure change in the second vessel

exhibits an initial pressure drop followed by a transition to an equalization pressure on a pressure vs. time curve. The envelope volume of the sample is then determined from a minimum pressure attained by the second vessel upon initial opening of the valve mechanism, wherein the minimum pressure is related to the initial pressure drop. The envelope density of the sample is then determined as a quotient of the mass and the envelope volume of the sample.

Another advantageous aspect of the present invention comprises a system for nondestructively determining a property of a porous sample having a mass. Such a system includes a first vessel capable of being evacuated to a sub-atmospheric pressure and a second vessel having the sample disposed therein and capable of being established at a test pressure, wherein the test pressure is greater than the sub-atmospheric pressure. A valve mechanism is operably engaged between the first and second vessels and is configured such that opening of the valve mechanism allows the pressures of the first and second vessels to equalize to an equalization pressure. A monitoring device is configured to determine a pressure change in the second vessel when the valve mechanism is opened, wherein the pressure change is indicative of a minimum pressure attained by the second vessel upon initial opening of the valve mechanism. The minimum pressure is related to an envelope volume of the sample, and thus a quotient of the mass and the envelope volume of the sample thereby provides an envelope density of the sample.

Thus, embodiments of the present invention provide a system and method capable of nondestructively determining the specific gravity, absorption, and other properties of soils, aggregates, and bituminous paving mixtures by utilizing the displacement of a gas in order to determine the volume of the sample. In addition, embodiments of the present invention provide a system and method capable of expeditiously producing the analysis of the sample, with minimal handling thereof, to obtain the necessary results with improved precision over commonly used methods. As such, embodiments of the present invention reduce the time necessary to perform the necessary sample analysis, while providing a higher degree of repeatability without contaminating or destroying the sample, thereby making the sample available for subsequent testing. Accordingly, embodiments of the present invention provide significant advantages as detailed herein.

BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWING(S)

Having thus described the invention in general terms, reference will now be made to the accompanying drawings, which are not necessarily drawn to scale, and wherein:

FIG. 1 is a schematic of a system for nondestructively determining a material property of a sample according to one embodiment of the present invention;

FIG. 2 is a schematic graph illustrating the pressure vs. time in the second vessel containing the sample as the valve mechanism is opened in order to equilibrate the pressure between then first and second vessels according to one embodiment of the present invention; and

FIG. 3 is a schematic graph illustrating the pressure vs. log (time) in the second vessel for the sample and a substantially nonabsorbent specimen, respectively, as the valve mechanism is opened in order to equilibrate the pressure between then first and second vessels according to one embodiment of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

The present invention now will be described more fully hereinafter with reference to the accompanying drawings, in which preferred embodiments of the invention are shown. This invention may, however, be embodied in many different forms and should not be construed as limited to the embodiments set forth herein; rather, these embodiments are provided so that this disclosure will be thorough and complete, and will fully convey the scope of the invention to those skilled in the art. Like numbers refer to like elements throughout.

FIG. 1 illustrates a system for nondestructively determining a property of a sample according to one embodiment of the present invention, the system being indicated generally by the numeral **10**. Such a system **10** comprises a first vessel **100**, a second vessel **200** configured to receive a sample **300** therein, a valve mechanism **400** in communication between the first vessel **100** and second vessel **200**, a gas source **500** and a vacuum source **600** both in communication with the valve mechanism **400**, and a monitoring device **700**. According to one advantageous aspect of the present invention, the system **10** is configured to determine the volume of, for example, a soil, aggregate, or bituminous paving mixture sample **300** and, in turn, the density and specific gravity

thereof so as to provide the necessary data required by applicable standards such as, for instance, ASTM Standard D2041, as previously mentioned.

More particularly, embodiments of the present invention utilize the ideal gas law of thermodynamics (PV=nRT) to determine the volume of a sample **300**, where P is the pressure, V is the volume, n is the number of moles of gas, R is a constant, and T is the temperature in degrees Kelvin. Determination of the volume of the sample **300** is accomplished by measuring the change in pressure as a gas is expanded from a closed second vessel **200** with a known volume, having the sample **300** disposed therein and established at a test pressure, into an evacuated first vessel **100** also having a known volume. As shown in **FIG. 1,** in order to accomplish the determination of the volume of the sample **300** (also referred to herein as "V_s"), the first and second vessels **100, 200** are sealable (and also referred to herein as "V_1" and "V_2", respectively), wherein the vessels **100, 200** may also be operably engaged with a device for monitoring the pressure therein such as, for example, a monitoring device **700** associated with the valve mechanism **400,** or a pressure gauge operably engaged with each vessel **100, 200.** The first and second vessels **100, 200,** in one embodiment, are comprised of aluminum due to, for example, thermodynamic considerations, as will be appreciated by one skilled in the art from the description herein of the applied methodology of the present invention. The gas source **500** is configured to provide a substantially inert gas such as, for example, helium, nitrogen, or carbon dioxide, while the vacuum source **600** comprises, for example, a vacuum pump. The gas source **500,** vacuum source **600,** and vessels **100, 200** are interconnected with appropriate tubing to one or more valves **410** comprising the valve mechanism **400.** The vessels **100, 200** are further optimized for expanding the gas from the second vessel **200** containing the sample **300,** as will be appreciated by one skilled in the art and as discussed further herein with regard to a propagation of error analysis for a system **10** as described.

Prior to the analysis of the sample **300,** the system **10** is first calibrated by, for example, performing a series of measurements, first with both vessels empty and then with a calibration object of known volume disposed within the second vessel **200.** The volumes V_1 and V_2 are then found, for a system configuration in which the sample **300** is

placed in V_2 and the gas is expanded from V_1 into V_1, so as to obtain an equalization pressure P_c, as follows:

$$V_1 = V_c \frac{\left(P_c P_o - P_o^2 - P_e P_c + P_e P_o\right)}{\left(P_e P_v - P_e P_o - P_c P_o + P_v P_c\right)}$$

$$V_2 = V_c \frac{\left(P_v P_o - P_e P_o - P_v P_c + P_e P_c\right)}{\left(P_e P_v - P_e P_o - P_c P_o + P_v P_c\right)}$$

$V_c \equiv$ volume of calibration object

$P_v \equiv$ evacuated pressure in V_1 prior to expansion

$P_o \equiv$ pressure in V_2 prior to expansion

$P_e \equiv$ expanded pressure in V_1 without calibration object

$P_c \equiv$ expanded pressure in V_1 with calibration object

5   Thereafter, in order to measure the unknown volume V_s of a sample **300**, the sample **300** is placed in V_2 and the gas is expanded into V_1 so as to attain the equalization pressure P_s. The volume V_s of the sample **300** is then determined according to the following relationship:

10   $$V_s = \frac{P_v V_1 + P_o V_2 - P_s\left(V_1 + V_2\right)}{P_o - P_s}$$

One skilled in the art will appreciate that the system **10** must be properly prepared prior to implementing the methods as detailed herein. For example, the system **10** may

be first evacuated using the vacuum source **600** acting upon the two vessels **100, 200** and the valve mechanism **400** via a connection to the valve mechanism **400**. Such a procedure removes or purges air from the system **10** as well as any water that may be present in the system **10** or the sample **300**. The application of the vacuum also causes

5   the vaporization of any water in the sample **300**, which facilitates removal of the water from the sample **300**. The vessels **100, 200** and the valve mechanism **400** may then be backfilled with the gas from the gas source **500** via a connection to the valve mechanism **400**. Such a vacuum and backfill procedure may be repeated as necessary, and monitored by the monitoring device **700**, in order to ensure that the vessels **100, 200** and valve

10   mechanism **400**, as well as the pores of the sample **300**, are essentially filled only with the gas. Upon completion of this purging process, the methods as described herein are accomplished in accordance with the presented underlying theories.

Embodiments of the present invention are particularly directed to determining the volume of a sample **300** using two vessels **100, 200** and a gas displacement methodology

15   according to the Ideal Gas Law. However, by this technique, there are two possible methods by which the volume of the sample **300** can be determined. As shown in **FIG. 1**, a sample **300** having an unknown volume is placed in the second vessel **200**. As such, one method of determining the volume of the sample **300** is to evacuate the second vessel **200** and pressurize the first vessel **100**, whereafter the vessels **100, 200** are connected and

20   the pressure allowed to equilibrate therebetween. A second method for determining the volume of the sample **300** is to evacuate the first vessel **100** and then pressurize the second vessel **200**, before equilibrating the pressure between the two vessels **100, 200**. The evacuated vessel may be evacuated to a pressure of, for example, about 20 Torr, while the pressurized vessel may be established at a pressure of, for example, about 700

25   Torr.

In comparing the two methods of determining the volume of the sample **300**, a propagation of error analysis was performed in order to determine which method provided the result with the highest precision. For each method, the equation for V_s was determined as a function of P_o (the positive pressure established in either the first vessel

30   **100** or the second vessel **200** prior to equilibration), P_s (the pressure following equilibration), the volume V_1 of the first vessel **100**, and the volume V_2 of the second

vessel **200**. Thereafter, the partial derivatives with respect to each of the independent variables were determined, multiplied by the respective uncertainty (s_x), and then added in quadrature. As will be appreciated by one skilled in the art, the result of the propagation of error analysis thereby provides an estimate of the total uncertainty in the measurement method as a function of V_1 and V_2 and V_s. The following are the partial derivatives as well as the equation for the total uncertainty.

Propagation of error for expanding from V_1 to V_2

$$Po \cdot V1 = Ps \cdot (V1 + V2 - Vs)$$

$$Vs = V1 + V2 - \frac{Po}{Ps} \cdot V1$$

$$\frac{d}{dPo}\left(V1 + V2 - \frac{Po}{Ps} \cdot V1\right)$$

$$\frac{d}{dPo}Vs = \frac{-1}{Ps} \cdot V1$$

$$\frac{d}{dPs}Vs = \frac{Po}{Ps^2} \cdot V1$$

$$\frac{d}{dV1}Vs = \frac{-(Po - Ps)}{Ps}$$

$$\frac{d}{dV2}Vs = 1$$

Propagation of error for expanding from V_2 to V_1

$$Po \cdot (V2 - Vs) = Ps \cdot (V1 + V2 - Vs)$$

$$Vs = V2 - \frac{1}{(Po - Ps)} \cdot Ps \cdot V1$$

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$$\frac{d}{dPs} Vs = -V1 \cdot \frac{Po}{(Po - Ps)^2}$$

$$\frac{d}{dPo} Vs = \frac{1}{(Po - Ps)^2} \cdot Ps \cdot V1$$

$$\frac{d}{dV1} Vs = \frac{-1}{(Po - Ps)} \cdot Ps$$

$$\frac{d}{dV2} Vs = 1$$

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$$s_{Vs} = \sqrt{\left(\frac{d}{dPo} Vs\right)^2 \cdot (s_{Po})^2 + \left(\frac{d}{dPs} Vs\right)^2 \cdot (s_{Ps})^2 + \left(\frac{d}{dV1} Vs\right)^2 \cdot (s_{V1})^2 + \left(\frac{d}{dV2} Vs\right)^2 \cdot (s_{V2})^2}$$

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A numerical minimization can then be performed for each of these cases resulting in an optimal ratio for the volumes V_1 and V_2 and a minimum uncertainty in the measured quantity. The results of this analysis indicated that the second method (expanding from V_2 to V_1) was slightly more precise having an uncertainty which was 5-

20   10% smaller than that of the first method, depending on the size of the sample **300**. Further, in one embodiment, an advantageous ratio of volumes V_1 and V_2 was found to be about 2:5.

The mass of the sample **300** is also determined such that, as a result, the density of the sample **300** may be obtained by dividing the mass of the sample **300** by the volume

25   V_s thereof. The density of the sample **300** may thus be expressed in units of, for example, g/cm^3, while the specific gravity of the sample **300** is determined as the unitless

ratio of the mass or density of the sample **300** to the mass or density, respectively, of an equal volume of water, both at the same temperature such as, for example, 25°C.

To obtain data with sufficient precision using a method and apparatus according to the present invention, an appropriate sample size should be used. For example, in the case of a bituminous paving material, the following sample size guidelines would be recommended:

Size of Largest Particle of Aggregate in Mixture, mm (in.)	Minimum Sample Size, g
50.0 (2)	6000
37.5 (1½)	4000
25.0 (1)	3000
19.0 (¾)	2000
12.5 (½)	1500
9.5 (3/8)	1500
4.75 (No. 4)	1500

In some instances, the capacity of the second vessel **200** may be limited. For example, in one embodiment, the second vessel **200** may have a volume V_2 of 2000 cm^3. In such instances, if the sample **300** has a mass greater than 2000 g, the sample **300** may be divided into two or more portions. Further, where the sample **300** has a mass of 6000 g, the sample **300** should be divided into three portions, each portion having a mass of 2000 g. In other instances, according to some embodiments of the present invention, a sample **300** should preferably have a mass of at least 1500 g.

A result of the described procedure is a density that is indicative of the absolute or apparent specific gravity (Gsa) of the sample **300**, as will be appreciated by one skilled in the art. However, the bulk specific gravity (Gsb) of a sample **300** of soil or aggregate, or the maximum specific gravity (Gmm) of a sample **300** of bituminous paving material, may also be determined. More particularly, the Gsb and Gmm parameters are skewed from the absolute or apparent specific gravity Gsa due to absorption of the gas by the sample **300**. Thus, according to one advantageous aspect of the present invention, the

pressure changes in the second vessel **200** (V₂) as the gas is expanded into the first vessel **100** (V₁) may be monitored as the gas absorbed by the sample **300** bleeds therefrom upon opening the valve mechanism **400** between the vessels **100, 200**. As a result, the envelope volume (as opposed to the absolute volume upon equilibration of the pressures in the vessels **100, 200**), and thus the envelope density, of the sample **300** may be determined, as described further below. The envelope density of the sample **300** is, in the case of soils and aggregates, the bulk specific gravity (Gsb), while, in the case of a bituminous paving material, the envelope density is the maximum specific gravity (Gmm). That is, the envelope density of the sample **300** is determined as the volume defined essentially by the outer surfaces of the sample **300** and includes any porosity within the sample **300**. The difference between the absolute (apparent) and bulk or maximum specific gravities are thus related to the absorption of the sample **300**, and may also provide an indication of the permeability characteristics thereof.

More particularly, when the valve mechanism **400** is initially opened to equilibrate the pressure between the two vessels **100, 200**, it has been found that the pressure in the second vessel **200** (containing the sample **300**), as experienced by the monitoring device **700** and as shown in **FIG. 2**, decays with a functional form equivalent to four coupled over-damped harmonic oscillators. At the same time, the gas absorbed in the sample **300** begins to diffuse or bleed from the sample **300**, which causes the pressure to rise to the equalization pressure **800** (Pₛ), the pressure rise having the functional form:

$$y = y_0 + a \cdot \left(1 - e^{-b \cdot x}\right) + c \cdot \left(1 - e^{-d \cdot x}\right)$$

As further shown in **FIG. 2**, the data between the minimum or base pressure **750** and the equalization pressure **800**, for example, the data between the first damped out harmonic oscillator and the equalization pressure **800**, may be used to extrapolate the trend of the data back to time t=0. Such an extrapolation back to time t=0 provides an indication of the theoretical minimum pressure **850** attained by the system **10** under a presumption that such a theoretical minimum pressure **850** would be attained if the valve mechanism **400** instantaneously opened and/or the system **10** allowed the vessels **100, 200** to be immediately connected before any of the gas could diffuse from the sample

300. Thus, the extrapolated theoretical minimum pressure **850** allows the envelope volume to be determined, the envelope volume of the sample **300** comprising the absolute volume of the sample **300** plus the volume of the pores therein. The envelope volume may thereafter be used to determine, for example, the theoretical maximum specific

5   gravity (Gmm) of a sample **300** of bituminous paving material, or the bulk specific gravity (Gsb) of a sample **300** of soil or aggregate, having that envelope volume. Accordingly, the absolute volume, which is determined from the final equalization pressure **800**, and the envelope volume, which is determined from the extrapolated theoretical minimum pressure **850**, can be used to determine the volume of the pores

10   within the sample **300** and, in turn, the absorption characteristics of the sample **300**. Further, the absorption characteristics of the sample **300** may be entered into a function describing the relationship between gas absorption and water absorption so as to determine the water absorption for the sample **300**.

       In some instances, the envelope volume determined from the theoretical minimum

15   pressure **850** is more accurate when the pores in the sample **300** are small. That is, for a sample **300** having small volume pores, the diffusion of the gas from the pores is relatively slow, and thus the theoretical minimum pressure **850** is sufficiently accurate for determining the envelope volume of the sample **300**. However, in instances where the sample **300** also includes larger pores (exhibits greater absorption), the diffusion of the

20   gas from these larger pores may be relatively fast and therefore the envelope volume of such a sample **300** may not be accurately modeled by the theoretical minimum pressure **850** alone. Such a sample **300** may be identified, for example, from a comparison of a pressure vs. log (time) curve for that sample **300** as compared to a pressure vs. log (time) curve for a substantially nonabsorbent specimen having about the same envelope volume

25   as the sample **300**, as shown in **FIG. 3**. A substantially nonabsorbent specimen may be comprised of, for instance, aluminum or the like, and will exhibit, for example, a faster pressure drop upon opening the valve mechanism **400** and a higher rate of equilibration to the final equalization pressure **800** than a sample **300** exhibiting absorbency (porosity). Thus, such a comparison between pressure vs. time curves for the sample **300** and the

30   substantially nonabsorbent specimen, respectively, may be manually performed by an

operator or, for example, by an appropriate computer device, and may also provide an indication of the relative absorbency of the sample **300**.

Many modifications and other embodiments of the invention will come to mind to one skilled in the art to which this invention pertains having the benefit of the teachings presented in the foregoing descriptions and the associated drawings. Therefore, it is to be understood that the invention is not to be limited to the specific embodiments disclosed and that modifications and other embodiments are intended to be included within the scope of the appended claims. Although specific terms are employed herein, they are used in a generic and descriptive sense only and not for purposes of limitation.